

ART I.—*Fixation of Phosphate in Some Victorian Soils.*

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Abstract.

Red soils on basalt in Gippsland give greater growth of pasture with lime and superphosphate than with superphosphate alone, on account of fixing phosphate in an unavailable adsorbed form. Various chemical extractions are recorded for these and for other soils which respond normally to superphosphate. The ratio of adsorbed to adsorbable phosphate is much lower for the high-fixing than for normal soils.

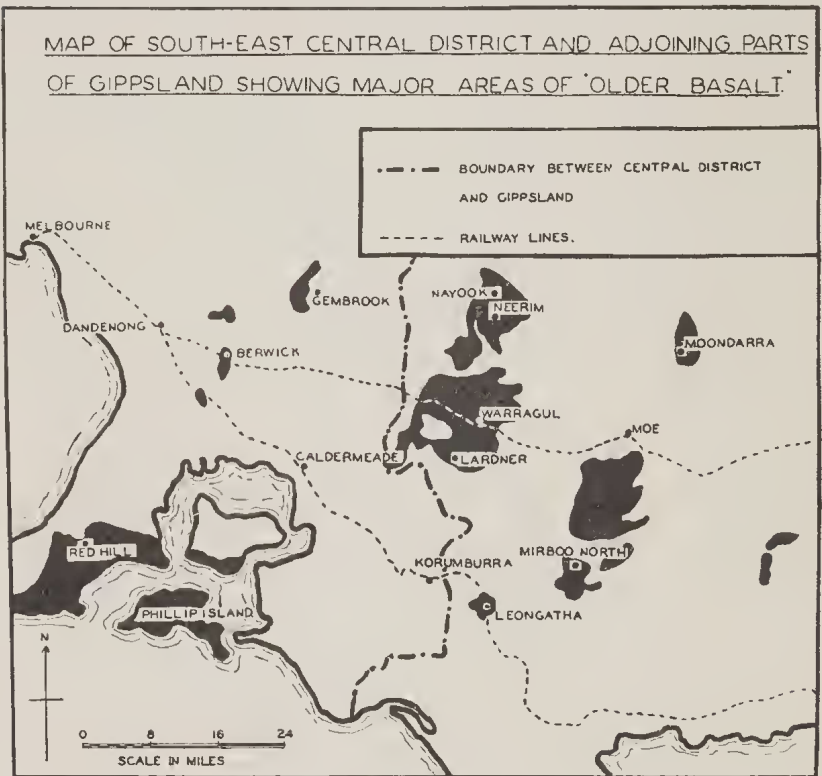
Most Victorian soils show evidence of phosphate deficiency which can be remedied by the application of superphosphate. An isolated group of deep red loams on oligocene basalt in South Gippsland near Mirboo North (see map), have shown a greater growth of pasture with lime and superphosphate, than with superphosphate alone; lime alone has little effect. This suggests that part of the added phosphate is being fixed by the soil in a form not available for plants, and that the addition of lime increases the availability of this phosphate. The term "fixation" has long been used to describe the conversion of soluble phosphate, added to the soil, into forms insoluble in water. In this paper the term is restricted to include only the conversion into forms not available for plants.

Soils occur in many parts of the world which fix soluble phosphate so firmly that plants can obtain little benefit from the application of superphosphate. These soils include both acidic and alkaline types; the present paper is concerned with acidic types only. The Hawaiian soils formed from basalt are the most widely quoted example of this problem. In Australia the most striking example is found on the basaltic country around Lismore, on the north coast of New South Wales. The climate of both these areas is warm and wet. The soils are friable and permeable, though high in clay; they are high in sesquioxides and red is the predominating colour. These are extreme cases in which added superphosphate has no effect at all. This conversion of phosphate into unavailable forms is slight in the red basaltic soils of South Gippsland, and is shown only by the additional response to lime and superphosphate mentioned above. Presumably the lime acts by reducing the activity of aluminium and iron compounds.

The literature on this subject is voluminous. In acid soils fixation in an unavailable form has been attributed to two constituents, free ferric oxide and kaolinite, which hold phosphate as an anion on their surfaces. Usually importance is attached

to the role of free ferric oxide in phosphate fixation, but Burd and Murphy (1) hold that kaolinite is mainly responsible, on account of the activity of the surface layer of alumina in its crystals.

The discussion of the problem by Burd and Murphy is particularly useful and is summarized and commented on in the following paragraphs. The analytical work reported here is a repetition of their methods, which were devised in order to estimate the phosphate status of the soils and to predict any future deficiencies. They chose 14 widely differing Californian soils, and obtained a good correlation between experimental results and field experience.



Absorbed Phosphate.

Burd and Murphy extract the soil with 0.1 N NaOH and thus determine the "adsorbed phosphate" by replacement with hydroxide. Dickman and Bray (2) suggest the use of neutral fluoride as a replacing ion since it does not dissolve organic matter. However, the small amount of organic phosphorus dissolved by alkali does not seriously increase the estimate of adsorbed phosphate.

The capacity of the soil to adsorb phosphate was next estimated by shaking a sample of soil with a solution of KH_2PO_4 , filtering and washing, and again determining the phosphate extracted by alkali. This figure is called the "adsorption capacity." The ratio of adsorbed phosphate to adsorption capacity, expressed as a percentage, is called the "degree of saturation." The higher this degree, the less firmly held and therefore the more available will any added phosphate be.

Burd and Murphy consider that it is useful to compare the "adsorption deficit"—that is, the difference between the two NaOH extractions—with the total phosphate. Thus Aiken clay, (see Table 1), which shows the most acute deficiency of their samples, has a far greater adsorption deficit than it has total phosphate. On the other hand, Yolo silty clay loam, which shows no phosphate deficiency, has a total supply of phosphate far above its adsorption deficit.

Unless the degree of saturation is high, adsorbed phosphate can only be available to plants by anionic exchange between the root hair surface and the soil particles. An extracting solution containing an anion of high replacing power should only be used for determining adsorbed phosphate, not for estimating available phosphate.

Dilute Acid Extracts.

The traditional line of attack is extraction with dilute acid. This can only give an approximate picture of the conditions existing between the plant and the soil. The plant obtains its phosphate by solution from soil particles due to an increase in acidity of the soil solution around the root. The best an acid extraction can do is to try to reproduce the simple acidity effects in the neighbourhood of the root hairs. Extraction with citric acid cannot do this since it also releases adsorbed phosphate. Phosphate adsorbed on the positive parts of soil colloids can be displaced by citrate ion, and although some plants may excrete organic anions which are strongly adsorbed, in general plants cannot use adsorbed phosphate unless the individual soil particles are themselves fairly well saturated with phosphate. Burd and Murphy describe the choice of citric acid as a solvent as "perhaps the most unfortunate occurrence in the history of soil phosphate investigation."

Hydrochloric acid comes close to the ideal since chloride ion has low displacing power. Burd and Murphy used 0.01 N HCl . The solution of sulphuric acid and ammonium sulphate recommended by Trnóg (12) is as high as $\text{M}/40$ in sulphate ion. It would therefore be expected to dissolve adsorbed phosphate more vigorously than dilute hydrochloric acid. Russell and Prescott (10, p. 89) found that sulphuric acid alone dissolved three times as much phosphate as did hydrochloric acid of the same normality.

Field Evidence in Victoria.

Most of the evidence of the phosphate status of the Victorian soils here described is given by experiments conducted by the Victorian Pasture Improvement League (V.P.I.L.) which has established numerous plots in southern districts to determine the best treatments with fertilizer. These plots are mowed periodically and the clippings weighed.

The superphosphate is applied annually at the rate of 2 cwt. per acre; (some plots receive an additional 1 cwt.); lime was only applied at the beginning of the experiments at the rate of 1 ton of calcium hydroxide per acre. This took place in 1935 for Lardner and Mirboo, and in 1932 for Korumburra, Caldermeade and Pakenham.

Superphosphate causes greater growth throughout the growing season of August to January. Any additional growth caused by lime is only obtained at the first spring cut in September (15, p. 216).

DESCRIPTION OF SOILS.

The soils tested are described below. They are referred to throughout this paper by their place names.

Red Soils.

Samples were taken from three sets of V.P.I.L. plots on the red soil formed on basalt near Mirboo North, in the hills of South Gippsland. The profiles of these are similar, viz. dark reddish brown friable clay loam at the surface, passing gradually to red friable clay at a depth of a foot. The soils are remarkably permeable in spite of the high proportion of clay. Solid rock is not usually found within several feet of the surface, but there may be occasional "floaters." The original vegetation was a heavy Eucalyptus forest, and particles of charcoal and baked clay are found in the surface soil, being residues of forest fires. The average annual rainfall is 45 inches.

Mirboo I. From Mr. Bickerton's property at Limonite. Application of lime caused a 50 per cent. increase over superphosphate alone in each of the years 1936-9. Organic carbon 4.1 per cent., pH 5.4.

Mirboo II. From Mr. Austin's property, Mirboo North. Lime caused 30 per cent. increase over superphosphate alone in the years 1936-7, and about 20 per cent. thereafter. Organic carbon 2.8 per cent., pH 5.8.

Mirboo III. From Messrs. Edney, Mirboo North. Lime caused no increase over superphosphate alone in 1936, then 30 per cent. increase each year 1937-39. Organic carbon 4.1 per cent., pH 5.8.

Samples were taken from two other places on the basalt near and north of Warragul.

Lardner (rainfall 40 inches) from V.P.I.L. plots on Mr. Teese's property. This soil is on the edge of the basalt and differs strikingly from the Mirboo soils. It is greyish brown with ironstone concretions from the surface downwards. It is deficient in potassium and has given no additional growth with lime. Organic carbon 3.8 per cent., pH 5.4.

Nayook (rainfall 48 inches). This soil is under Eucalyptus forest. The profile is essentially similar to the Mirboo samples. Organic carbon 4.2 per cent., pH 5.5.

A further sample was included from the Lismore district, New South Wales, since this type has been intensively studied by Holman (5) and Parbery (8).

Wollongbar (rainfall 52 inches). Red friable clay loam, overlying red friable clay, with weathered basalt at about 5 feet. No response to lime has been established here. The fixing power for phosphate is very high. Organic carbon 3.4 per cent., pH 4.0.

Grey and Black Soils.

A sample developed on Oligocene basalt was taken from Berwick (Vic.), where both reddish-brown and black soils are formed on this same parent material.

Berwick (rainfall 34 inches). Black friable clay overlying greyish black heavy clay, with decomposing rock at 2 feet (6, p. 192). Subsoil much less permeable than the red types. No evidence of added growth with lime. Organic carbon 4.2 per cent., pH 5.6.

The remaining samples are taken from other V.P.I.L. plots. They show no response to lime, but are included for interest as they lie near the red soils of Mirboo under a similar climate.

Korumburra (rainfall 46 inches). The surface soil is a grey clay loam with a yellowish grey clay at 12 inches, which passes into decomposing rock which is a felspathic sandstone of Jurassic age. This is an immature podzolic type. Organic carbon 2.9 per cent., pH 5.3.

Caldermeade (rainfall 30 inches). Dark grey clay loam, passing into heavy clay at 8 inches. The land is flat and low-lying and has been described as "swamp fringe" type by Goudie (3). Organic carbon 4.2 per cent., pH 5.0.

Pakenham (rainfall 34 inches). Light grey sandy loam, overlying yellow sandy clay at 18 inches. This is a well-marked podzol similar to Harkaway sand (6, p. 187). Organic carbon 2.5 per cent., pH 5.5.

The mean annual temperature for the Victorian stations is close to 57°F.; rainfall reaches a maximum in winter and early spring. At Wollongbar the mean annual temperature is 67°F.; the rain falls mainly in summer and autumn.

All the surface soils in the Victorian samples are from 0-3 inches, subsurface from 3-12 inches. The surface sample from Wollongbar is from 0-9 inches.

The two Californian soils already referred to have been thus described (16).

Aiken clay is a somewhat lateritic soil developed on basalt in rolling country. The surface is a dull red clay which passes gradually into the subsoil which is a more pronounced red, compact and with a heavier texture, being high in clay. Both soil and subsoil are mildly acidic in reaction and contain aggregates or concretions cemented by sesquioxides of iron and manganese and with accumulated phosphate in an insoluble and unavailable form.

Yolo silty clay loam is a deep immature light coloured alluvial soil, alkaline in reaction, rich in lime and mineral plant nutrients, but low in organic matter.

ANALYTICAL METHODS.

Phosphate in Adsorbed Condition.

Thirty gram of soil was shaken for 1 hour with 150 c.cs. 0.1 N NaOH. The suspension was centrifuged and the supernatant liquid was poured off, diluted with an equal volume of 0.1 N NaOH, and filtered through a porcelain candle. An aliquot of 20 c.cs. of the black filtrate was boiled with 15 c.cs. concentrated sulphuric acid in a Kjeldahl flask, adding 5 gm. $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ and 1 gm. MnSO_4 crystals as catalyst. The colourless product was diluted, filtered from silica, and made up to 100 c.cs. with distilled water. An aliquot of 5 or 10 c.cs. was neutralized with ammonia using α -dinitrophenol as indicator, and the phosphate determined colorimetrically.

Adsorption Capacity.

Thirty gram of soil was shaken for 15 hours with 150 c.cs. of KH_2PO_4 solution containing 3 mg. PO_4 per cc. The soil was filtered and washed with distilled water until 1500 c.cs. had passed through. It was then shaken with 150 c.cs. 0.1 N NaOH, and treated in the same way as the previous soda extract. When determining the adsorption capacity of soils after the removal of free ferric oxide (see below), the suspension after extraction with soda was flocculated with sodium sulphate, instead of being filtered through the candle.

This method yields a lower figure for adsorbed phosphate than would be given by the alternative method of simply determining the phosphate left in the solution of KH_2PO_4 after coming to equilibrium with the soil. The difference between the two methods is not very serious. Burd and Murphy say that they leached out "free phosphate" in the soil with distilled water. However,

even after prolonged washing the soil still yields some phosphate to distilled water and the above method has been used in order to remove the most lightly held fraction.

Acid-soluble Phosphate.

Thirty gram soil was shaken intermittently with 150 c.cs. $\cdot 01$ N HCl for 24 hours. The temperature of the suspensions was kept close to 20°C . during the day.

Before the phosphate in the extract could be determined colorimetrically, organic matter and ferric iron had to be destroyed. The extract was evaporated to dryness and treated successively with 1 c.c. concentrated hydrochloric acid containing a drop of nitric acid and hydrogen peroxide prepared by mixing sodium peroxide and sulphuric acid. The residue was left to stand overnight with an acid solution of sodium sulphite to destroy ferric iron. The phosphate was then determined colorimetrically.

Colorimetric Determination of Phosphate.

The method used (reduction of phosphomolybdate) was based on that given by Truog and Meyer (14). The aliquot was neutralized in a 50 c.c. standard flask with ammonia, using *a*-dinitrophenol as indicator. The volume was made up to about 40 c.cs., 2 c.cs. molybdate solution added (a 2.5 per cent. ammonium molybdate solution in $10\text{N-H}_2\text{SO}_4$); then 5 c.cs. diluted stannous chloride solution, and the volume made to 50 c.cs. The blue colour was compared with the standard after 10 minutes.

A 3.75 per cent. solution of hydrated stannous chloride in N HCl was stored under paraffin and diluted with water (1:10) as required.

The sodium hydroxide and ammonia solutions were stored in waxed bottles to avoid contamination by silicate which also forms a blue compound under these conditions.

Phosphate Soluble in Concentrated Hydrochloric Acid.

The soil was boiled for 2 hrs. with concentrated hydrochloric acid, and the phosphate determined gravimetrically as phosphomolybdate.

Free Ferric Oxide.

This was found using the method of Truog et al (13) viz. extraction with Na_2S at pH 7 followed by acidification with oxalic acid.

Organic Carbon.

This was determined by the approximate volumetric method of Tiurin (11).

DISCUSSION OF RESULTS.

Dilute Acid Extracts.

The amounts extracted by dilute acid (Table I. col. 6) are very low compared with the American figures except that for Aiken clay, which appears to be of the same order as the red soils from Mirboo. Plots of the red soils which have been annually topdressed with 2 cwt. per acre of superphosphate for the last five years give similarly low figures, whether treated with lime or not. The uncultivated forest soil from Nayook gives a strikingly low figure.

TABLE I.—PHOSPHATE STATUS OF VARIOUS SURFACE SOILS.

All Australian samples are 0-3 inches except Wollongbar, 0-9 inches.

Locality or Soil Type.	PO ₄ per 100 gm. Soil.		Present Degree of Saturation.	PO ₄ per 100 gm. Soil.		PO ₄ Soluble in 0.1 N HCl.	Field Evidence of Phosphate Status.
	Adsorption Capacity.	In Adsorbed Condition.		Adsorption Deficit.	Dissolved by Conc. HCl.		
	mg.	mg.	%	mg.	mg.	p.p.m.	
Mirboo I. ..	173	26	15	147	116	1.2	20-50 per cent. increase with superphosphate. Lime + superphosphate, 50 per cent. above superphosphate alone
Mirboo II. ..	161	18	11	143	72	0.7	Over 50 per cent. increase with superphosphate. Lime + superphosphate, small additional increase
Mirboo III.	192	16	8	176	100	1.0	100-200 per cent. increase with superphosphate. Lime + superphosphate, 30 per cent. above superphosphate alone
Nayook ..	246	3	1	243	56	0.1	Forest
Lardner ..	173	11	6	162	122	0.7	50 per cent. increase with superphosphate only after adding potassium
Wollongbar	308	19	6	289	920	1.3	Pasture not improved by phosphate with or without lime. Symptoms of deficiency observed.
Korumburra	165	60	36	105	108	1.2	Nearly 100 per cent. increase with superphosphate. No improvement with lime.
Caldermeade	106	47	45	59	130	5.6	50-100 per cent. increase with superphosphate. No improvement with lime
Pakenham ..	90	26	39	64	48	1.7	Big increase with superphosphate
Berwick ..	52	15	29	37	64	2.3	Increased growth with superphosphate
Aiken clay	547	22	4	525	160*	2.0†	Extreme phosphate deficiency
Yolo silty clay loam	55	27	48	28	200*	91	No phosphate deficiency

* Fusion analyses.

† Burd and Murphy give this figure as 0.2 mgm. per cent. (p. 335). It is probably a misprint for <0.2. Murphy on page 351 of the same issue records all acid extracts of Aiken clay as being <0.2 though he apparently did not determine the actual amount present.

When working with a soil from Lismore similar to Wollongbar, Holman (5) found that over the pH range of 8 to 2 both native and added phosphate were completely insoluble.

The grey soils of Korumburra and Caldermeade, after ten years of topdressing with 3 cwt. superphosphate per acre, show increases in extractable phosphate; viz. from 1.2 to 4.0 and from 5.6 to 7.5 parts per million respectively. The black soil at Berwick developed on the same parent material as the red Gippsland soils has a higher figure for soluble phosphate.

The pH values of the acid extracts of the unlimed samples were either within or close to the range 4.0-4.5; the limed plots gave extracts 0.5-1 pH unit above the unlimed.

Table 2 shows that the sub-surface horizons contain much less phosphate in the adsorbed condition or dissolved in dilute acid. This would be expected, and confirms the general opinion that the top few inches of soil are the most important.

TABLE 2.—PHOSPHATE STATUS OF SUBSURFACE HORIZONS (3-12 INCHES).

Locality.	In Adsorbed Condition.	Extracted by .01 N. HCl.
Mirboo I.	5	0.1
Mirboo II.	8	0.3
Mirboo III.	8	0.4
Nayook	0.1	0.1
Korumburra	13	0.5
Caldermeade	14	0.2

Adsorbed Phosphate.

Dealing next with adsorbed phosphate, the most important feature of Table 1 is the figure for percentage saturation, which shows a good correlation with experience in the field. This figure is high for the grey or black soils (Korumburra, Caldermeade, Berwick, Pakenham) which respond normally to superphosphate; and for Yolo silty clay loam, which naturally yields an ample supply of phosphate to plants. It is low for the red soils. Of these, the soils of Mirboo are recorded as giving additional response to superphosphate when lime is added. Lardner gives no such response to lime. An acute deficiency in potassium has been found there, and this may be a complicating factor.

Aiken clay is very deficient in phosphate, but Burd and Murphy give no indication of the effect of applied superphosphate or lime.

The soil from Wollongbar is peculiar. It is very high in total phosphate, but has a low percentage saturation, and yields little phosphate to dilute acids or in the Neubauer test (8). The pastures, consisting mainly of *Paspalum*, give no indication of response to superphosphate with or without lime. This soil may be similar to Hawaiian soils quoted by Heck (4) where the fixing power is so intense that liming to pH 6.5, which he calls the point of maximum solubility of the phosphate, causes only a slight improvement.

Free Ferric Oxide.

Free ferric oxide (Table 3), while lower in the red soils of Gippsland than in the Wollongbar sample, is higher than in the neighbouring grey soils such as Caldermeade. In the black sample from Berwick, however, it is actually higher than in the red soils of Mirboo.

TABLE 3.—FREE FERRIC OXIDE AND ITS EFFECT ON ADSORPTION CAPACITY.

Locality.	Free Ferric Oxide.	Adsorption Capacity PO ₄ per 100 gm. Soil.	
		Before Removing Fe ₂ O ₃ .	After Removing Fe ₂ O ₃ .
	%	mgm.	mgm.
Mirboo I.	4.9	173	13
Nayook	3.2	246	13
Lardner	2.9	173	7
Wollongbar	11.2	308	22
Korumburra	2.7	165	n.d.
Caldermeade	1.0	106	n.d.
Pakenham	1.7	90	n.d.
Berwick	5.2	52	n.d.

The adsorption capacity of the soils after the removal of free ferric oxide is very low. It must be concluded that in the soils studied, the greater part of phosphate fixation takes place on the surface of free ferric oxide.

Silica-sesquioxide Ratio.

Any study of the fundamental nature of the problem of phosphate fixation should include a knowledge of both the chemical composition and physical structure of the colloid in the soils taken. This has not been attempted, but the following figures for silica-sesquioxide ratios of similar soils are of interest.

Prescott and Hosking (9) give a $\frac{\text{SiO}_2}{\text{R}_2\text{O}_3}$ ratio of 0.7 for their sample from Wollongbar; this was much the lowest ratio of all the red basaltic soils from Queensland and New South Wales

which they studied. A common figure for this group of soils was 1.4. Red soils on basalt near Berwick have a ratio of 1.7 (6, p. 234). It is likely that the red Mirboo and Nayook soils are similar.

Staining Tests.

Lundblad (7) has suggested an approximate method of finding the colloidal properties of soils, by measuring the amounts of methylene blue and alizarin R which the soil can adsorb. The amount of each dye adsorbed depends on the amount and nature of colloidal matter in the soil. The cation of methylene blue is adsorbed onto the negative part of the colloid (silicate or humate) and the anion of alizarin R onto the sesquioxides. The ratio of $\frac{\text{cation adsorbed}}{\text{anion adsorbed}}$ thus gives a measure of the silica-sesquioxide ratio.

Table 4 shows the results obtained following Lundblad's method using alizarin S instead of alizarin R.

TABLE 4.—CAPACITY OF SOILS TO ADSORB METHYLENE BLUE AND ALIZARIN S.

Locality.	Methylene Blue Adsorbed by 1 gm. Soil.	Alizarin S. Adsorbed by 1 gm. Soil.	Relative Adsorbing Power $\frac{\text{Col. 1.}}{\text{Col. 2.}}$
	mg.	mg.	
Mirboo I. ..	52	15	3.5
Lardner. . .	42	17	2.5
Wollongbar ..	20	17	1.2
Korumburra ..	38	12	3.2
Caldermeade ..	66	8	8.2
Pakenham ..	22	4	5.5
Berwick ..	80	16	5.0

The siliceous soils of Caldermeade, Berwick and Pakenham have high ratios. Wollongbar, as expected, is low, Mirboo, Lardner and Korumburra intermediate. Soils with a relatively low ratio and a high capacity for adsorbing alizarin S, would be expected also to have a high adsorption capacity for phosphate. This does not mean that all these soils have a high fixing capacity for phosphate, as they already may be fairly saturated, e.g. Korumburra. The low ratio for Korumburra can be correlated with its high ferric oxide content.

CONCLUSION.

In conclusion, it appears that the analytical methods suggested by Burd and Murphy will be useful in diagnosing the state of phosphate in the field; and as more figures are obtained, their usefulness should increase.

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